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Ultrahigh-pressure experiment with a motor-driven diamond anvil cell

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Abstract
A Pt sample was compressed to ultrahigh pressures in a diamond anvil cell (DAC) using a motorized gearbox to change pressure remotely from outside the synchrotron x-ray hutch. *In situ* angle-dispersive x-ray diffraction (XRD) was used to determine pressure from known equations of state (EOS). The sample position was unperturbed during motor-driven pressure changes. By eliminating the need to realign the sample to the x-ray position after each pressure increment, 142 XRD patterns could be collected continuously over the course of three hours, and the maximum pressure of 230 GPa was reached before diamond failure ended the experiment. We demonstrate the advantages of this motor-driven assembly for smooth and efficient pressure change, and the possibility for fine pressure and temporal resolution.

1. Introduction

High-pressure science is a rapidly expanding research area that spans a myriad of different fields—physics, chemistry, materials science, earth science, and bioscience. The physical and chemical properties of materials can be altered radically as they are compressed, leading to the discovery of new phenomena and phases. The diamond anvil cell (DAC) is the only tool for reaching ultrahigh (>60 GPa) static pressures with a temperature range of 35 mK to 6000 K. A large array of microscopic techniques using laser, x-ray, neutron, and electromagnetic probes is available for studying the minute samples *in situ* at high pressure and temperature. For instance, synchrotron x-ray diffraction can be used to determine the structure and volume of the sample for investigation of phase transitions and equations of state (EOS).

In a DAC, the anvils are typically compressed mechanically by springs through a screw and lever arm arrangement [1], or pneumatically by diaphragm [2] or membrane [3]. Relative to a pneumatic system, the mechanical screw-spring system has the advantages of: (1) stability without the concern of gas leaks (a cell has been kept at constant pressure at 200 GPa since

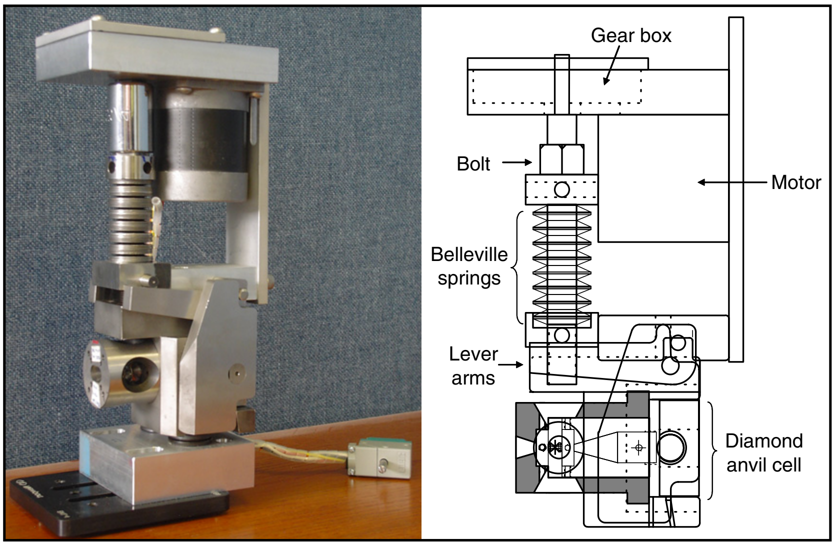

Figure 1. Motorized diamond anvil cell assembly: left, picture; right, mechanical drawing. (This figure is in colour only in the electronic version)

1986), (2) ease of transportation under high pressures, (3) low-temperature operation unlimited by the freezing point of the pneumatic gas, (4) accommodating large displacements without the rupture of membrane, and (5) simplicity and low cost, without the attachment of a gas bottle and control. A pneumatically controlled membrane, on the other hand, has the advantages of the remote and quantitative control of force increment without having to dismount or disturb the DAC. These advantages, however, can also be achieved in a mechanical system if the screws are driven by a motorized gearbox instead of the ordinary manual operation. The ability to increase the pressure remotely in a mechanical DAC would greatly improve measurement efficiency, which in turn would enable strain-rate control, increase the accuracy of EOS determination, help in pinpointing the exact point of phase transition, etc.

We modified the spring–screw mechanism by attaching a motor-driven gearbox to the system, and achieved high stability with remote-control operation. Here, we present our experimental result in compressing Pt to 230 GPa using a motorized mechanical bolt–spring–lever arm arrangement. We were able to take many measurement points without moving the DAC. This was due to the stability of the system, which eliminated the time-consuming steps of scanning the sample to check the position after each pressure increment.

2. Experimental details

The experiment was carried out in a piston–cylinder type of DAC. The piston–cylinder assembly was mounted in a double lever arm, which was driven by a spring-loaded 1/2-inch bolt with 20x mechanical advantage. A gearbox with an additional 20x mechanical advantage was added to this conventional spring–screw–lever arm DAC, and was driven by a step motor for changing pressure (figure 1). The double lever arm and motor-driven gearbox system can be used universally for compressing different varieties of DAC, such as a symmetrical DAC for double-sided laser heating [4] and the panoramic DAC for side XRD and x-ray spectroscopy [5].

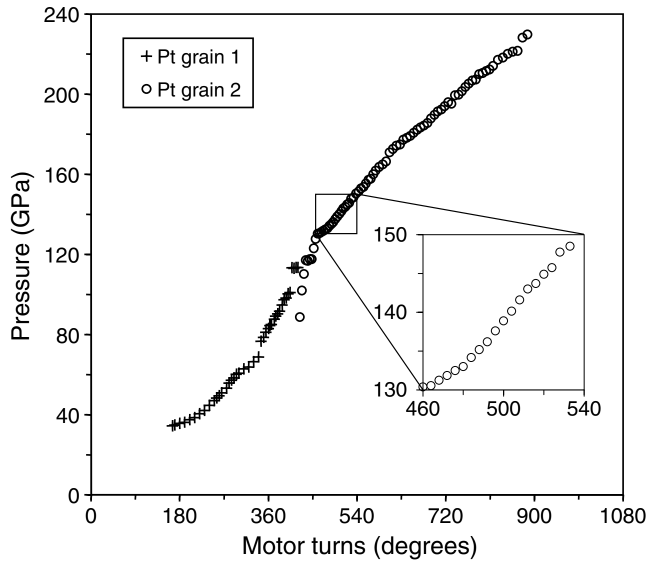

Figure 2. Relationship between motor turns and pressure measured on Pt in the DAC sample. Inset shows magnified view over a smaller range, which illustrates the fine and smooth pressure gradations possible with this system.

Two bevel anvils are used: the anvil on the cylinder side had a 300 μm outer culet diameter, 100 μm centre flat diameter, and 10° bevel, and the anvil on the piston side had 300 μm outer culet diameter, 40 μm centre flat diameter, and 9° bevel. A 30 μm hole was drilled in an Re gasket into which a small piece of Pt was added.

Monochromatic synchrotron radiation ($\lambda = 0.4246$ Å) at the 16 IDB undulator beamline of the High Pressure Collaborative Access Team (HPCAT), Advanced Photon Source, Argonne National Laboratory was used for angle-dispersive x-ray diffraction measurements. The incoming x-ray was focused by a pair of bimorph Kirkpatrick–Baez mirrors to 10 μm $\times$ 16 μm full width half maximum (FWHM) and directed through the diamond axis, and the diffracted beam was collected with a MAR CCD detector (exposure time 30 s), and the powder diffraction images were processed using the Fit2d software [6].

Sixteen stainless-steel Belleville spring washers, each with dimensions of 12.83 mm I.D. (inner diameter), 25.4 mm O.D. (outer diameter), 1.27 mm thickness, and 1.91 mm height, were packed in a two-parallel, eight-in-series arrangement, and compressed by a 1/2-inch diameter screw with 1/2-20 thread to generate the load. With an individual spring constant of 3.8–5.1 kN mm^{-1}, turning the bolt provided 53–71 N/degree force on the lever arm or 1.1–1.4 kN/degree on the sample, and turning the 20:1 gearbox using the motor provided the next load on the sample. These values agree with the pressure/load estimate for DAC at 100–300 GPa [7], while accurate pressure determination depends on internal x-ray calibrations [8].

Pressure was increased remotely in 4° steps of motor rotation. The volume of the Pt sample was measured after each step and the corresponding pressure was determined from a known Pt EOS [8] (figure 2). We measured 142 x-ray diffractions over a 3 h period with dense sampling (every 1–2 GPa) over a large pressure interval (40–230 GPa). The experiment terminated when one of the diamond anvils shattered at the maximum pressure of 230 GPa, which represented 2.5 turns (900°) of the gearbox.

3. Discussion

Ultrahigh pressures in the DAC are generated by building up a steep stress gradient across the diamond culet in solid samples and sampling the very small central peak area of maximum compression and minimum gradient [9]. One common practice for searching for this peak area is by conducting two-dimensional step scans of the DAC position relative to the small x-ray beam (see [9]); first, a fast scan over a larger range ($\pm 100\ \mu$m) of the DAC sample stage in the two orthogonal directions normal to the x-ray beam while monitoring the sample's absorption of x-rays to locate the peak position within 10 μm and, second, a slow scan over the range of $\pm 10\ \mu$m while collecting the x-ray diffraction pattern at each step to pin down the peak-pressure area within 1–2 μm. A partial or complete search procedure can take 10–60 min. Manually turning the screws to increase pressure often causes a slight shift of the DAC position, which necessitates a very time-consuming peak area search. It also requires an additional 5–10 min to open the hutch, increase pressure manually, search the hutch, and close the hutch door. Each pressure increment step normally takes at least 15–20 min. This severely restricts the efficient usage of synchrotron beam time and prohibits large data-set collection at small pressure intervals, thus limiting accurate determinations of the EOS and phase transition boundary. Moreover, time-dependent information, associated with the first few seconds to minutes after pressure increase, is lost.

These problems are eliminated by the remote motor-driven pressure increase, which only needs 1 s to turn the motor while leaving the sample position undisturbed, and several seconds to collect a diffraction pattern. The advantages were demonstrated in the present experiment. Approximately halfway into the experiment (at a motor rotation 420°–430°), we scanned the DAC position and mapped the pressure distribution in the Pt grain, which covered a pressure range from 90 to 120 GPa in a distance of 15 μm (the large spike in figure 2), showing a gradient of 2 GPa μm^{-1}. Further increasing the load remotely using the motor produced a very smooth pressure versus motor rotation plot; the deviation from a smooth curve is <1 GPa, indicating that the disturbance of the sample position by such an operation is $<1\ \mu$m. Other noticeable kinks of 2–3 GPa in the curve were associated with long time breaks (10–15 min) between consecutive pressure increments, and are attributed to plastic deformation and relaxation of the gasket. By removing the temporal overhead and positional disturbance in the pressure change operation, the time-dependent change of strains in the gasket and the sample can be monitored to provide information on viscoelastic flow. In addition, many P–V points can be recorded instantaneously for EOS determination.

Another application of the motor-driven DAC is in the area of moderate pressure for studies of biological materials [10] or clathrates [11]. These experiments do not need to go beyond 200 MPa, but require fine resolution within the pressure range. The pressure resolution of ruby fluorescence or internal x-ray diffraction calibrations, typically ± 50 MPa, can be a large fraction of the entire pressure range and are inadequate. For a DAC with a 1 mm diameter, flat culets that are typically used in moderate-pressure studies, and a 2° rotation of the present motor-driven system will deliver the load of 1 MPa pressure increments, thus providing the desirable fine control.

4. Conclusions

New developments and improvements in technology play a large role in the continued growth of high-pressure science. Here, we demonstrate the use of a motorized mechanical bolt–spring–lever arm system for remote pressure increase up to the maximum pressure capability of the DAC. The system has many potential applications. For instance, the extreme stability of the

DAC position allows for rapid, efficient x-ray diffraction measurements and enables time-dependent strain-rate measurement after a remote increase of stress.

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